

Synthesis, Characterization, and Application of Waste Material Made Magnetic Nanoparticles for the Recovery of Adulterated Markings on Metallic Surfaces

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Serial numbers are marks present on several day-to-day objects, created by manufacturers for purposes such as quality control, product tracking and to prevent counterfeiting. In the case of firearms, these characters can be obliterated or damaged by natural processes or intentionally by criminals to prevent positive identification of stolen property. One non-destructive method employed by crime laboratories for the restoration of obliterated characters is the magnetic particle method. The present work aims to study the synthesis and application of waste-made magnetic nanoparticles in the recovery of adulterated markings. A set of analytical-grade materials and metallic waste magnetic nanoparticles were synthesized for comparison purposes. Chemical characterization tests by X-ray fluorescence, morphological tests by scanning electron microscopy (SEM), and magnetization tests by vibrating sample magnetometer (VSM) were performed. The efficacy of the synthesized nanoparticles was tested on 1020 steel plates previously engraved and adulterated by sanding. Both homemade magnetic nanoparticles demonstrated superior performance compared to commercial magnetic particles. The application of homemade magnetic particles, especially using waste as the source of production, decreases the cost of the material and is environmentally friendly. The development of this material makes magnetic serial number restoration a better option for widespread use in forensic procedures.

Keywords: nanoparticle, sustainability, magnetic restoration, serial number, waste

Introduction

Serial numbers or product identification numbers are present on many everyday objects, such as computers, medical implants, automobile parts, and firearms. Other applications include jewelry, industrial machinery, motorcycles, and medicine packaging.¹ Manufacturers use serial numbers for purposes of quality control, product tracking, product liability, and to prevent counterfeiting. The numbers may conform to a standard, such as the Universal Product Code, or may be unique to the manufacturer. In most instances, they are simply a sequential series of numbers and/or letters that describe how many units in a particular series have been produced, sometimes giving

information related to the brand, model, caliber, or other specific information. Serial numbers are often deleted by criminals to avoid the tracking of stolen or illegally acquired products such as firearms and cars.²

One of the main activities within forensic chemistry laboratories is restoring the serial numbers on firearms and vehicle chassis. There are different ways to restore a serial number,³ usually associated with the composition of the metallic plate, how it was originally printed, and the obliteration method applied by the criminals. For many decades, the most popular method of serial number application was stamping, which involves compressing the characters into the metal surface using direct force. Other application methods include casting and engraving. A serial number could be obliterated or damaged by a natural process, such as corrosion, exposure to extreme environments, or, more commonly, manual and intentional

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obliteration by criminals usually using chemical or physical processes. Misapplication of a restoration method can destroy the evidence before proper identification jeopardizing the investigation. In forensic chemistry laboratories in Brazil, only two methods of revealing serial numbers are frequently used: the fry method and the electrochemical method.⁴ Both methods are based on a controlled corrosion process in which the damaged area presents a different electrochemical potential than the surrounding and may be selectively attacked at a different rate.⁵ Deformed regions of the marking are more chemically active and, therefore, will corrode at a higher rate than unaffected regions. Thus, a visible contrast will be created due to the reflection or differential dispersion of light in the areas of residual tension, making it possible to reveal the original marking.

Despite the broad applicability of both methods, they are still considered destructive methods as they result in a non-reversible corrosion of the metallic evidence. Additionally, these methods are not able to reveal the serial numbers from all cases submitted to forensic laboratories due to their limitations.^{2,6,7} Alternative methods for recovering serial numbers include heat treatment, hardness profiling, ultrasonic cavitation, backscattered electron diffraction, and visualization by refraction or reflection of X-rays. These methods present high costs and are limited by the need of specific equipment not always available for cases where the serial numbers need to be revealed in crime scenes or in big pieces of evidence such as cars.¹

A method for restoring damaged serial numbers using magnetic particles has been applied in forensic laboratories but with limited success. Its advantage relies on the fact that it is a non-destructive method. The success of the technique depends on many factors such as the use of an appropriate magnetic field, its orientation, and the choice of the ideal magnetic particle. Other factors such as the properties of the metallic surface and the degree of aggressiveness to remove the serial number must also be considered.⁸ The magnetic particle method is based on the dispersion of the magnetic particles over the surface of the serial number and the subsequent application of a magnetic field. In those materials where the inscriptions have been damaged, the visible parts have been removed, but the deformations in the metal remain. The deformation site can cause a “leakage” in the magnetic flux and attract the particles, revealing the original numbers.^{9,10}

Virtually all forensic labs in the United States use the product named Magnaflux[®] when performing magnetic restoration of serial numbers.⁷ This product uses magnetic iron oxide powder to reveal the markings. Although there are different versions of this product (in oil suspension

or powder, different colors, and fluorescent ones), all the versions of this iron oxide powder are produced in bulk size using iron-derived raw materials.⁷ Iron oxide nanoparticles possess an important role in this method due to their superparamagnetic nature, their small size, and their wide range of applications. Despite the popularity of Magnaflux[®] in the U.S., this product is not available in Brazil due to the elevated cost. Alternatively, iron oxide magnetic nanoparticles could be produced from cheaper materials.

The use of metallic waste material for the synthesis of magnetic nanoparticles decreases the overall cost and minimizes the environmental impact of its production. This waste is formed during the process to produce steel in steelworks. Iron is obtained from the reduction of iron oxide with coal and carbon monoxide in a blast furnace in the presence of fluxes (limestone, silica, or dolomite). This mixture reacts to produce pig iron (the intermediate product of iron ore smelting) and some impurities such as powders, sludge, and slag. The carbon formed as an impurity is transferred to a steel mill where the injection of oxygen converts it into CO and CO₂, transforming the pig iron into steel. A dust-type residue containing SiO₂, iron, and other solid particles is deposited to the walls of the steelwork facility forming a solid waste that is usually removed and discarded by the steel industry. This waste can be treated and used for the synthesis of magnetic iron nanoparticles.^{11,12}

This work aimed to develop a synthetic process to produce magnetic nanoparticles from steelwork waste and evaluate its application in the recovery of serial numbers and marking in forensic evidence. For that, magnetic nanoparticles were produced using steelwork waste, and laboratory-grade materials. The synthesis followed the procedure described by Chang *et al.*¹³ involving the reduction of iron oxide by ammonium hydroxide under heating and agitation. After cooling to room temperature, the supernatant was discarded. Both materials were characterized by energy dispersive X-ray fluorescence, and the magnetic properties of the nanoparticles were measured by vibrating sample magnetometry. Both homemade nanoparticles had their efficacy in recovering markings evaluated and compared to the commercial magnetic particle performance popular in forensic laboratories.

Experimental

Reagents

Iron(III) chloride hexahydrate (FeCl₃·6H₂O) and ammonium hydroxide (NH₄OH) were obtained from Merck KGaA (Darmstadt, Germany). Mohr's Salt (iron(II)

ammonium sulfate hexahydrate, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was obtained from Labsynth (Diadema, São Paulo, Brazil). Water utilized was double-distilled and deionized in a water purification system (Sartorius, Darmstadt, Germany). The solid metal waste was obtained from a local steel industry (Ternium, Santa Cruz, Rio de Janeiro, Brazil). The commercial magnetic particles Supermagna[®] YD 404 used for comparison studies were obtained from Metal Check (Bragança Paulista, São Paulo, Brazil). The isopropyl alcohol was acquired by Reagen (Rio de Janeiro, Brazil).

Magnetic nanoparticle synthesis

The homemade magnetic nanoparticles were produced using laboratory-grade reagents and steel waste using two distinct methods. For the laboratory-grade synthesis, the magnetic nanoparticle production was performed according to the procedure described by Chang *et al.*¹³ For that, 3.05 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 2.9 g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ were dissolved in 50 mL of water, mixed, and then heated at 90 °C. Then, 10 mL of an ammonium hydroxide 25% (m/v) solution was quickly added to the mixture. The black solution formed was mixed for 30 min at 90 °C and then cooled to room temperature. The mixture was centrifuged at 4000 rpm for 10 min and the black deposit formed was collected and washed with water five times.

The synthesis of the nanoparticles from the metallic waste followed the same procedure previously applied to the laboratory-grade reagents,¹³ with an additional pre-treatment step. The pre-treatment consisted of the addition of 60 g of the waste material to a 500 mL beaker containing 100 mL of water. The mixture was agitated for 30 min and left to rest to decant the solid. The supernatant was discarded and 100 mL of a 0.1 mol L⁻¹ solution of nitric acid was added to the solid. The mixture was left in agitation for 30 min and the separation process was repeated. The solid material was then washed again in water followed by a neutralization with NaOH 0.1 mol L⁻¹. A final water washing was performed to remove the excess base. Third grams (30 g) of the pretreated solid were then used for the synthesis of the magnetic nanoparticle as previously described.

Characterization of the nanoparticles

The chemical characterization of the homemade magnetic nanoparticles was performed on an EDX 7200 energy dispersive X-ray fluorescence spectrometer (Shimadzu, Kyoto, Japan). Morphology studies were performed on a JSM-7100F thermal field emission electron microscope (Jeol, Akishima, Japan). To prepare the samples for microscopy, the particles were dispersed in an isopropyl

alcohol solution, subjected to ultrasound for approximately 30 min and dropped onto conductive carbon tapes. The magnetic properties of nanoparticles were measured on a vibrating sample magnetometer VersaLab 3 Tesla Cryogen-free (Quantum Design, San Diego, CA, USA).

Test specimen

For the evaluation of the performance of the nanoparticles, 1020 steel sheets (steel for tools) obtained from Gravamaster[®] (Mauá, São Paulo, Brazil) and TCM Co. (Talladega, AL, USA) were used as test specimens. Steel sheet serial numbers were previously recorded by punching with a hammer with the defined model with numbers and letters (AWB 124) in a depth of 0.3 mm and height of 5 mm. The markings were obliterated using the sanding method.

Recovery of markings using the magnetic nanoparticles

The test specimens were magnetized using a Y-6 YOKE electromagnet (Magnaflux[®], Glenview, IL, USA) using a 1.8 A current and 50/60 Hz of frequency. The magnetic particles were applied over the metallic surface using the wet method. For that, isopropyl alcohol was used as the solvent and the solution was prepared at a concentration of 9 g L⁻¹ of magnetic nanoparticles.

A small amount of the emulsion containing the magnetic nanoparticle in isopropyl alcohol was added to the pre-treated specimen. The electromagnet was used to magnetize the plate in both directions, making 90° of each application, as suggested by the standard procedure for the recovery of serial numbers using magnetic particles.¹⁴ For each assay, a photographic register was performed. At the end of each assay, the test specimen was cleaned with sandpaper and methanol. A total of 10 trials with each magnetic particle were performed. The images obtained were converted into scores as described in Table 1.

Magnetic nanoparticles performance scale

The evaluation of the performance of the magnetic nanoparticles was based on the observation of the obliterated numbers after the recovery process. A successful recovery requires the numbers to be clear and easy to read (high neatness). The proposed scale for the visual comparison of the efficiency of the magnetic particles is shown in Table 1. For each visualization profile, a score was given based on the deposition of the material in each inscription and the capacity to read the characters with high confidence. To define the values of each score, each assay was registered by digital pictures in ten replicates.

Table 1. Scores obtained after restoration using the magnetic method

Score	Observation
0	there is no restoration of the mark
1	part of the alfa numeric characters seems to be restored, but not enough to identify no one character
2	part of the alfa numeric characters are restored, enough to know at least one character
3	most of the alfa numeric characteristic are restored, enough to know all characters even if the image is not 100% restored
4	total visualization of all hidden characters

The results for both homemade magnetic nanoparticles were compared to the commercial nanoparticles. Statistical analysis was performed using the software GraphPad Prism 9¹⁵ and IBM SPSS Statistics 27.¹⁶

Results and Discussion

Chemical and magnetic properties of the nanoparticles

The chemical composition of the homemade magnetic nanoparticles and the commercial product using X-ray fluorescence spectrometry are shown in Table 2. The results show that the nanomaterial produced through chemical-grade reagents is composed almost entirely (98.9%) of iron oxide.¹⁷ The commercial particle also presented a composition with a high percent of iron oxide (99.1%) although with a different composition predominating FeO and Fe₂O₃. The nanoparticle produced using waste material showed a similar composition to the one prepared with laboratory-grade materials differing only with the presence of residual compounds such as Si and calcium oxides that are commonly present in steel waste.

Morphological characteristics of the particles obtained by emission electron microscopy are shown in Figure 1. The nanoparticles produced with laboratory-grade reagents (Figure 1a) presented a size between 50 and 300 nm, waste nanoparticles (Figure 1b) presented a comparable size, in the 100-300 nm range, and the commercial particles (Figure 1c) presented higher size, size about 4 μm. The images show agglomerated particles due to their magnetic nature, which was enhanced when preparing the materials for analysis, which were dispersed in isopropyl alcohol.

The magnetic properties of the particles were measured by vibrating sample magnetometry (VSM). The operation of a magnetometer consists of the generation of a magnetic field by coils in the region where the sample is located, which, after being magnetized, will produce an induced field that is read by the sensor.¹⁸ The magnetization curve relates the external magnetic field applied to the sample

Table 2. Chemical composition of the magnetic material obtained by X-ray fluorescence spectrometry analysis. For this analysis, the X-ray fluorescence spectrometer was operated using air atmosphere, collimator at 5 mm, and mylar sample cup

Element	Synthesized through chemical grade reagents / (% m/m)	Synthesized through metal waste / (% m/m)	Commercial / (% m/m)
Fe	45.4	23.5	34.3
O	53.4	68.1	64.8
Ca	0.031	6.3	N.D.
Si	0.31	1.0	0.33
S	0.41	0.24	0.17
Mn	0.18	0.30	0.12
Zn	0.09	0.30	0.013
K	N.D.	0.055	N.D.
Sr	N.D.	0.011	N.D.
Bi	N.D.	0.007	N.D.
Cu	N.D.	0.005	0.005
Pb	N.D.	N.D.	0.011

N.D.: not detected.

with the magnetization induced by it in the sensor, where the saturation magnetization (Ms) is the value where all the magnetic moments of the sample are aligned with the external magnetic field, representing a maximum in the magnetization curve verified by the formation of a plateau on the graph. The external magnetic field that resulted in this saturation magnetization value is called the saturation field (Hs), which means that the magnetization from this field value will remain constant.¹⁹

Magnetic nanoparticles (MNPs), when subjected to an external magnetic field, present better magnetization saturation values (Ms) in relation to larger magnetic particles. Furthermore, these values are reached more quickly, as they require a smaller saturation field (Hs). Magnetic nanoparticles still have a more uniform magnetization, because unlike larger magnetic particles where there are several regions of uniform magnetization, in MNPs this smaller particle size results in only a single region of uniform magnetization, causing all spins to be aligned in the same direction and the particle is magnetized uniformly.²⁰

The results are shown in Figure 2 and indicate that the nanomaterials produced with chemical reagents and waste are basically composed of magnetite (Fe₃O₄), since the magnetization saturation value (Ms) was around 60-80 emu g⁻¹.²¹ A slightly higher value of the saturation magnetization (Ms) of the particles synthesized with waste can be attributed to the larger average size of their nanoparticles.²² The presence of impurities in the waste

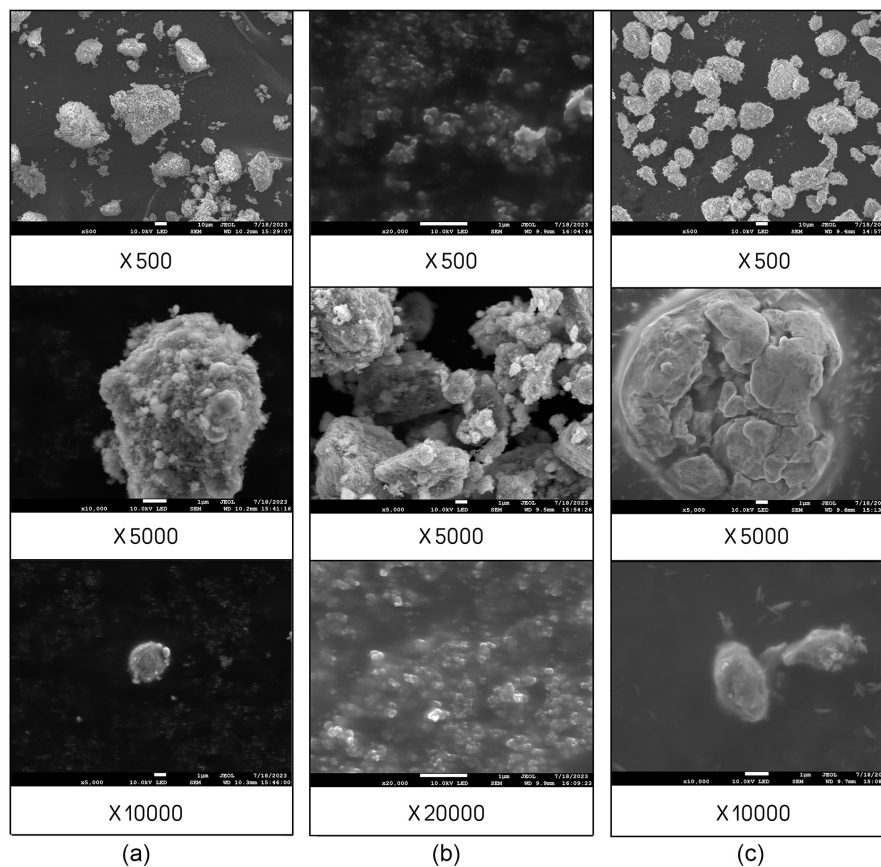


Figure 1. SEM image of magnetic particles (a) laboratory-grade nanoparticles, (b) waste magnetic nanoparticles, (c) commercial magnetic particles with a magnification between 500 \times (up) and 3000 \times (below).

sample, with a greater proportion of calcium oxide (CaO), increased the saturation field (H_s).²³ Commercial particles have a magnetization saturation value (M_s) compatible with a mixed phase (hematite and magnetite) since hematite has typical saturation magnetization values (M_s) in the order of 1-5 emu g^{-1} .²⁴

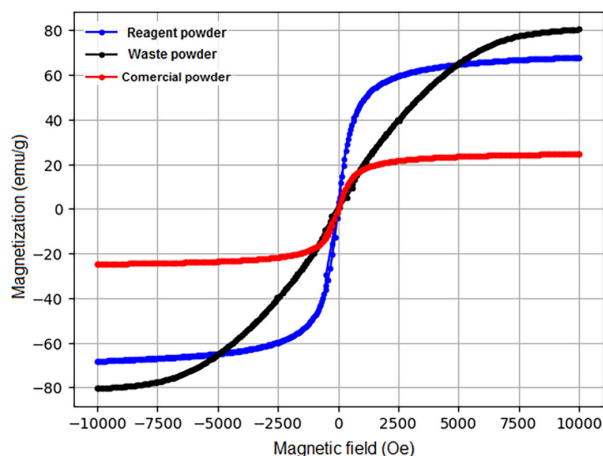


Figure 2. Hysteresis loops of magnetic particles measured at 300 K. The magnetization of the iron oxide nanoparticles *versus* the magnetic field at 300 K was obtained by cycling the field between -20 and 20 kG.

The amount of iron, particle quality, and application method (wet method) are characteristics that influence the performance of the analysis. Very small particles have greater sensitivity for detecting minor discontinuities but require a better finish on the part, as they can be trapped in surface irregularities.²⁵ The serial number magnetic recovery procedure applies electromagnets with a low magnetic field (around 80 Oe),^{26,27} thus, a better result is expected when using nanoparticles synthesized with laboratory-grade reagents as it has a higher magnetization saturation value (M_s) in a lower saturation field (H_s), requiring a smaller external magnetic field to magnetize all particles in the same direction when compared to waste nanoparticles and commercial particles, in which similar low-field performance is expected. The nanoparticles synthesized with waste even have the highest saturation magnetization value, but the field value necessary to achieve this magnetization is very high and not compatible with the equipment that will be used in the tests.

Magnetic performance of the nanoparticles

For the analysis of the performance of the three

nanoparticles tested, the serial number magnetic recovery procedure was applied. Inscriptions were adulterated using the sanding method. As the magnetic procedure is non-destructive, the test specimen was evaluated several times for statistical purpose. The appearance of the test specimen in different steps of the procedure is shown in Figure 3. The scores obtained after ten trials for each nanoparticle are shown in Table 3.

Table 3. Scores of the test with magnetic particles

Measurement	Score		
	Commercial particles	Nanoparticles synthesized with reagents	Nanoparticles synthesized with metal waste
1	1	4	0
2	0	4	2
3	1	4	2
4	2	3	2
5	0	2	2
6	0	2	2
7	0	2	2
8	0	4	1
9	2	4	1
10	0	3	2

The scores obtained by the visual analysis of each set of ten trials for each nanoparticle were applied for the statistical comparison of the efficiency of each nanoparticle. The normality of the data set was evaluated using both Kolmogorov-Smirnov and Shapiro-Wilk tests. The results showed that variables laboratory-grade nanoparticle (LB), waste nanoparticle (WS), and commercial nanoparticle (CP) presented a normal distribution (LB, $K-S(10) = 0.128$, $p < 0.001$; $S-W(10) = 0.945$, $p < 0.001$; WS, $K-S(10) = 0.128$, $p < 0.001$; $S-W(10) = 0.945$, $p < 0.001$; CP, $K-S(10) = 0.128$, $p < 0.001$; $S-W(10) = 0.945$, $p < 0.001$).

After the normality of the distribution of the results was evaluated, an analysis of variance²⁸ was applied to test

whether there was a difference between the means of the results. An F calculated of 25.2 was found for a critical value of 3.35, suggesting a statistical difference in the mean value.

After this, a paired t -test was made between each pair of results and a statistical difference was found in every means. The result of this study is in accordance with the images shown in Figure 3. This demonstrates that the magnetic nanoparticles synthesized with reagents were the ones that had the best results in the recovery of the digits “1” and “2”. The nanoparticles synthesized with metallic waste had good results in the number “1” but in relation to the number “2” which was partially removed, the result was not as good as the nanoparticles synthesized with reagents. Commercial magnetic particles were not able to recover the inscriptions satisfactorily.

It shows that the nanomagnetic material produced through chemical grade reagent was the best magnetic material produced, and even the magnetic material produced through waste material presented behavior better than the commercial material, showing that this work presents exciting options for replacements of the magnetic material available in the market.

Conclusions

In this work, two types of nanomagnetic particles based on iron oxide were produced: one using chemical-grade reagents, and the other using iron oxide waste from the steel industry. The particle production procedure was virtually the same for both, and these materials were used in the recovery of adulterated markings on metallic surfaces through the technique of magnetic particle serial number restoration. To evaluate the efficiency of these materials, the same procedures were carried out with commercial magnetic particles. The results showed that, using the recommended procedure and a commercial magnet, the chemical-grade reagent-based material was the most efficient, the waste-based material was second,

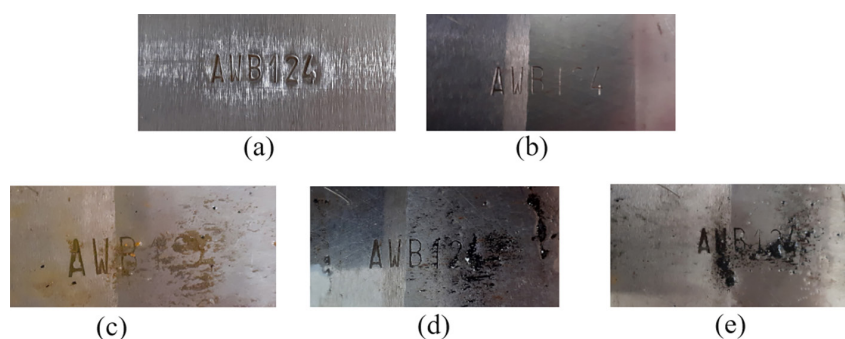


Figure 3. Test specimen before being obliterated (a), after (b), and after the treatment with the different magnetic materials: (c) commercial magnetic particle, (d) laboratory-grade nanoparticle and (e) waste nanoparticle.

and both were better than the commercial one. The characterization by X-ray fluorescence was important to determine the chemical composition of each material. Morphological characterization confirmed the production of nanoparticles, which were smaller in size compared to commercial particles, while characterization by vibrating sample magnetometer demonstrated that the produced nanoparticles have greater magnetization, especially within the magnetic field values achieved by commercial magnets. This material testing can assist criminal investigations as an alternative method for identifying adulterated numbers, with the added advantage of being non-destructive.

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Author Contributions

Daniel B. Souza was responsible for conceptualization, formal analysis, investigation, methodology; Bruno G. Silva for data curation, investigation; Rubem L. Sommer for data curation; Iane A. Soares for data curation; Gabriela R. Pereira for conceptualization, data curation, investigation; Rafael M. Dornellas for conceptualization, data curation, investigation, project administration; Wagner Felipe Pacheco for conceptualization, data curation, funding acquisition and investigation.

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